

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

PATENT SPECIFICATION

NO DRAWINGS

1142377

1142377



Date of Application and filing Complete Specification: 6 Oct., 1966.

No. 44752/66.

Application made in United States of America (No. 502576) on 22 Oct., 1965.

Complete Specification Published: 5 Feb., 1969.

© Crown Copyright 1969.

Index at acceptance:—A5 B(15, 32)

Int. Cl.:—A 61 k 7/16

COMPLETE SPECIFICATION

Chewable Material

We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is performed, to be particularly described in and by the following statement:—

This invention relates to a chewable material for use in cleaning the teeth.

A chewable material according to the present invention comprises 15 to 25% by weight of a water-soluble protein, a vegetable gum, and a liquid polyhydric alcohol in which the gum and protein are soluble at a temperature in the range 50° to 90° C; there being present approximately equal weights of the protein and gum, and the weight of alcohol present being approximately twice the weight of the protein.

The chewable material is a chewable mass of cohesive resilient edible material soluble in saliva and is substantially free of fermentable carbohydrates the mass is non-adherent to the teeth, and, during chewing, gives a rubbery yielding resistance to the closing of the jaws and deforms in response to the pressure exerted by the teeth; the mass slowly dissolves and breaks into small particles within a few minutes under the continued influence of saliva and repeated chewing.

When the mass is bitten between the molars during chewing, it becomes deformed and thus comes into contact with many of the tooth surfaces, even between the teeth, without breaking up into fragments. Since it is not adherent to the tooth surface it comes away freely from the teeth during chewing, to be reformed by tooth pressure the next time the jaws are brought together. The size of each unitary mass is such that in initial chew-

ing the teeth encounter a relatively thick mass of the material which has a rubbery, yielding resistance to the closing movement of the jaws; the unitary mass yields and its outer surface becomes deformed without significant cutting of its outer surface by the cusps of the teeth. A great deal of the deformation of the material on biting is elastic, that is, the mass is indented by the cusps of the teeth but the indentation disappears to a large extent after each bite owing to the elasticity or resilience of the material. As chewing proceeds, the outer portions of the unitary mass dissolve away gradually in the saliva, its thickness decreases and its surface becomes less resistant to cutting, so that the unitary mass tends eventually to dissolve entirely or to break up into small fragments which can be swallowed as such, chewed or sucked. This fragmentation does not occur, however, until after the mass has been chewed vigorously for more than half a minute, generally more than a minute.

Advantageously, the weight of the unitary mass is in the range 1 to 10 grams, preferably 3 to 5 grams. In the preferred products, the unitary mass has at least one dimension in the range 3.2 mm to 19.0 mm, more preferably 9.5 to 15.9 mm. In a preferred form, the mass is compact and not extended in any particular direction for example, its shape may be like that of a cube, a sphere, or a right cylinder, (e.g. having a ratio of height to diameter of 1:1 to 2:1), although the material may be produced in other less regular shapes. Also, the unitary masses need not be dispensed to the user in entirely discrete form; for example, a rod of the material of the desired diameter may be weakened at spaced points along its length, as by cutting partially through the rod at regular intervals, so that individual, unitary masses can be torn easily off the end of the rod by the user.

[Price 4s. 6d.]

The unitary mass may include a sialogogue to stimulate the flow of saliva, which helps to remove food particles from the teeth. For this purpose, any of the well known substantially non-fermentable sweetening agents and flavouring agents, including breath freshening agents, may be employed.

The chewable material of this invention has particular utility for cleaning the teeth and freshening the mouth after meals in situations where tooth brushing is impracticable or impossible. It has been found that the chewing of the material of this invention results in extensive removal of trapped food particles, and greatly reduces the level of fermentable carbohydrate material (which is known to contribute to caries formation) in the mouth. The composition, being completely edible, leaves no solid residue. The mouth is cleaned and freshened quickly and easily.

Preferably the water-soluble protein is gelatin, the vegetable gum is gum acacia and the liquid polyhydric alcohol is glycerine. Best results have been obtained by using a gelatin having a high bloom number (a well known measure of the penetrability of a standard set solution of the gelatin in water); Bloom numbers above 150, advantageously at least 190, e.g. above 230, most preferably in the range 265 to 285, are preferred. In the preferred compositions some water is also present.

As already indicated, the protein and the gum are present in approximately equal weights e.g. in a weight ratio within the range of 1.2:1 to 1:1.2, while the weight of polyhydric alcohol is approximately twice the weight of protein e.g. about 2.2 times the weight of protein). The proportions of the polyhydric alcohol and water may be varied in accordance with the resiliency desired; in the preferred compositions the amount of (water is approximately equal to the amount of protein e.g. about 0.8 or 0.9 times the amount of protein), so that the weight of water and polyhydric alcohol is approximately three times the weight of the protein. In general, the proportions of the ingredients will be within the following ranges: protein 15 to 25%; gum 12.5% to 30%; polyhydric alcohol 30 to 55%; water 12 to 25% total water from all components.

In the absence of the gum, a protein-polyhydric alcohol compositions would tend to have little cohesiveness and to break up readily into many small particles on chewing. The preferred compositions also contain a water-insoluble finely divided dental polishing agent. Preferably, the dental polishing agent is an abrasive material having an average particle size below 10 microns. A particularly suitable polishing agent is finely divided water-insoluble sodium metaphosphate (e.g. having an average particle size of 2 to 10 microns). Other polishing agents to assist in removal of film from the teeth are alu-

minas, chalks, calcium phosphates, aluminium silicates and zirconium silicate. The proportion of polishing agents may be, for example, up to 20%, e.g. in the range 2 to 20%, based on the weight of the total mixture. It is found that the presence of the finely divided polishing agent acts as a reinforcing filler resulting in an increase in the resistance to penetration of the material.

Other ingredients which are advantageously incorporated into the composition are small amounts of non-fermentable sweetening agents such as sodium cyclamate, calcium cyclamate, saccharine, or combinations of sweetening agents; preservatives for the protein, such as sodium benzoate; germicidal or anti-bacterial agents such as quaternary ammonium compounds, e.g. benzethonium chloride, (di-isobutylphenoxyethoxyethyl dimethyl benzyl ammonium chloride), cetyl pyridinium chloride, lauryl dimethyl benzyl ammonium chloride, or other non-toxic germicidal agents, such as hexachlorophene. Preferably, the composition includes a flavouring agent as well as a colouring agent; thus, there may be employed various synthetic flavours, e.g. essential oils which provide fruit flavours, e.g. grape, lemon, lime, or orange, mint flavours or spice flavours (e.g. cinnamon flavour) all of whose compositions are well known. To help solubilize the germicide and colour and avoid any tendency for these components to precipitate, it is sometimes desirable to add a dispersing agent such as polyoxyethylene sorbitan monooleate.

In one useful method for the preparation of the preferred composition, the gelatin, gum and finely divided polishing agent are dispersed in the glycerine at room temperature. At this stage, the mixture is smooth and paste-like, containing fine solid dispersed particles of the gelatin and gum. The other ingredients are dissolved in the water and the water and glycerine mixtures are blended to form a very fluid mixture which is then heated, preferably to a temperature in the range 50—90° C (e.g. 74° C), whereupon the gelatin dissolves and the mass thickens greatly. The amount of gum acacia is generally such that complete solution does not take place in the presence of the other ingredients. The flavouring agent is then added to the molten mixture, which is thereafter shaped and cooled, as by pouring into moulds of suitable size. It is advantageous to carry out the entire mixing operation under vacuum so as to produce a product substantially free of gas bubbles. It has been found that the presence of dispersed air decreases the cohesiveness of the resulting unitary masses and also makes the molten material more resistant to flow during the shaping operation. The use of 610—740 mm of vacuum throughout the process has given excellent results.

The best properties of the material are

70

75

80

85

90

95

100

105

110

115

120

125

130

obtained at predetermined water contents. To avoid altering the water content, it is advantageous to pack the unitary masses in suitable sealed containers. The unitary masses may be wrapped individually in moisture-resistant barrier materials such as aluminium foil or "Saran" (trade mark) film.

The following Examples illustrate the invention. In the Examples and elsewhere all percentages are by weight unless otherwise indicated.

EXAMPLE 1

A composition was produced, using the technique described above, by mixing the following ingredients: glycerine, 39.20%; gelatin (Type A, i.e. acid type gelatin; Bloom 277; viscosity 50.5 millipoises, determined on a standard 12.5% aqueous solution at 60° C, as specified by the National Association of Glue Manufacturers, United States of America) 22.41%; gum acacia 20.98%; water-insoluble sodium metaphosphate (of average particle size 4.8 ± 0.5 microns) 4.00%; added distilled water 10.99%; 1% solution in water of FD & C Red No. 2, 0.90%; 1% solution in water of FD & C Red No. 4, 0.10%; benzethonium chloride 0.05%; sodium benzoate 0.01%; sodium cyclamate 1.12%; flavour (double mint: e.g. peppermint plus spearmint) 0.24%.

The composition was formed into cylindrical masses 17.5 mm in diameter and 12.7 mm high having rounded bases.

EXAMPLE 2

A composition was produced, using the technique described above, from the following ingredients: glycerine 37.03%; gelatin (as in Example 1) 18.77%; gum acacia 17.58%; water-insoluble sodium metaphosphate (as in Example 1) 12.55%; added distilled water 10.09%; sodium cyclamate 1.12%; citric acid monohydrate, which augments the flavour 1.00%; sodium citrate dihydrate 1.5%; sodium benzoate 0.01%; synthetic lime flavour 0.15%; 0.25% solution in water of F D & C No. 1, 0.08%; 1% solution in water of F D & C Yellow No. 5, 0.12%. The composition was moulded into the same shapes as in Example 1.

EXAMPLE 3

A composition was produced, using the technique described above, from the following ingredients: glycerine 39.62% (including 0.27% of water based on the total composition); gelatin (as in Example 1) 19.49% (including 1.95% of water based on the total composition); gum acacia 18.25% (including 2.55% of water based on the total composition); water-insoluble sodium metaphosphate as in Example 1) 10.00%; sodium benzoate 0.01%; sodium cyclamate 1.21%; citric acid monohydrate 1.00%; sodium citrate dihydrate 1.50%; plus water and colour solu-

tion. The total amount of water, as determined by analysis of the finished composition, was 15.00%. The composition was shaped into cylinder 12.7 mm in diameter and 19.0 mm long.

The preferred compositions are homogeneous and strongly cohesive but do not have substantial tack. Thus when cut or torn apart and pressed together again (at 25° C) they do not adhere strongly. Their adhesion is even less when their surfaces are wet by saliva or water; such wetting makes their surfaces become slick or slimy. However, if the surfaces are moistened and then permitted to dry partially, they become tacky. Also, at very low temperatures (e.g. -40° C) the masses have been found to tend to fuse together when brought into contact. The masses become softer as the temperature is raised. Their melting points are usually in the neighbourhood of 43-49° C; when melted on a hot stage the material does not tend to flow readily but is very sticky and strings out when it is touched with an instrument and the instrument is pulled away. The masses become softer as the temperature is raised, e.g. to 37.8° C. Their specific gravity is usually in the neighbourhood of 1.3.

One indication of the physical properties of the material is its resilience. This may be measured, for example, on an "Instron" tester which exerts tension on a specimen at a predetermined rate, then releases the tension and records the results, automatically plotting a graph of elongation (as abscissa) vs. tension (as ordinate). Two curves are recorded, one representing the behaviour of the material under tension and the other its recovery towards its original dimensions (a so-called "hysteresis loop" being thus produced). The area under the first curve represents the work required to stretch the specimen the predetermined distance and the area under the second curve represents the work performed by the specimen in recovering. Dividing the second area by the first gives a "resilience index". In one series of tests, a cylinder of the material of 12.7 mm diameter and 19.0 mm long was pierced diametrically by two parallel 2 inch standard steel finishing nails spaced 6.3 mm apart; the nails were then attached, respectively, to the two pulling arms of the "Instron" tester. The arms were then moved apart 3.05 mm to stretch the specimen by that distance and then returned to their original position. Both movements were effected at a rate of 5.08 mm per minute. In typical specimens of material of the invention, the resilience index, so determined, was well over 0.5, e.g. above 0.7, for instance in the range of 0.74 to 0.77. The force necessary to extend the specimen the 3.05 mm distance was generally over 300 grams, e.g. 350-410 grams. The measurements were made at room temperature. It was also found that the speci-

mens recovered their original length during the test, without any substantial permanent set.

- 5 Another measure of the physical properties of the material is its penetrability. This may be determined on a standard Precision penetrometer using a 150 gram weight on the penetration needle and allowing the weighted needle to rest on the surface of the material
10 for five seconds. The values of penetrability so determined will vary depending on the type of needle and the shape of the specimen. Using a thin nail as the needle and applying it to the flat upper surface of a cylindrical
15 unitary mass having a diameter of 17.5 mm and a height of 12.7 mm and having a rounded base, penetration values below 6 mm, e.g. 3.5 to 5 mm, were obtained.

WHAT WE CLAIM IS:—

- 20 1. A chewable material for cleaning the teeth comprising 15 to 25% by weight of a water-soluble protein, a vegetable gum, and a liquid polyhydric alcohol in which the gum and protein are soluble at a temperature in
25 the range 50° to 90° C; there being present approximately equal weights of the protein and gum, and the weight of alcohol present being approximately twice the weight of the protein.

2. A chewable material as claimed in Claim 1 in which the protein is gelatin, the vegetable gum is gum acacia and the polyhydric alcohol is glycerine.

3. A chewable material as claimed in Claim 1 or Claim 2 in which the amount of gum present is in excess of the amount which is soluble in the remainder of the material.

4. A chewable material as claimed in any of the preceding claims which also contains water and in which the weight of water and polyhydric alcohol is approximately three times the weight of the protein.

5. A chewable material as claimed in any of the preceding claims which also contains a water-insoluble finely divided dental polishing agent.

6. A unitary mass of chewable material as claimed in any of the preceding claims and weighing 3 to 5 grams.

7. A unitary mass as claimed in Claim 6 having at least one dimension in the range 3.2 to 19.0 mm.

8. A chewable material substantially as described in any of the Examples.

KILBURN & STRODE
Chartered Patent Agents
Agents for the Applicants

THIS PAGE BLANK (USPTO)